

# REPORT DOCUMENTATION PAGE

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| 1. REPORT DATE (DD-MM-YYYY)  |                             |                              | 2. REPORT TYPE<br>Technical Papers     |                     | 3. DATES COVERED (From - To)                                   |  |
| 4. TITLE AND SUBTITLE<br><br><i>Please see attached</i>  |                             |                              |  |                     | 5a. CONTRACT NUMBER  |  |
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| 9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)<br><br>Air Force Research Laboratory (AFMC)<br>AFRL/PRS<br>5 Pollux Drive<br>Edwards AFB CA 93524-7048 |                             |                              |  |                     | 10. SPONSOR/MONITOR'S ACRONYM(S)                               |  |
|  |                             |                              |  |                     | 11. SPONSOR/MONITOR'S NUMBER(S)<br><i>Please see attached</i>  |  |
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| 13. SUPPLEMENTARY NOTES  |                             |                              |  |                     |  |  |
| 14. ABSTRACT   |                             |                              |  |                     |  |  |
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| 15. SUBJECT TERMS  |                             |                              |  |                     |  |  |
| 16. SECURITY CLASSIFICATION OF:  |                             |                              | 17. LIMITATION OF ABSTRACT<br><i>A</i> | 18. NUMBER OF PAGES | 19a. NAME OF RESPONSIBLE PERSON<br>Leilani Richardson          |  |
| a. REPORT<br>Unclassified  | b. ABSTRACT<br>Unclassified | c. THIS PAGE<br>Unclassified |  |                     | 19b. TELEPHONE NUMBER<br>(include area code)<br>(661) 275-5015 |  |

MEMORANDUM FOR PRS (In-House Publication)

FROM: PROI (STINFO)

11 May 2001

SUBJECT: Authorization for Release of Technical Information, Control Number: AFRL-PR-ED-VG-2001-115  
Mario Fajardo and Michelle DeRose, "Status of Cryosolid Propellants Task"

**AFOSR Molecular Dynamics Contractors' Meeting**  
**(Irvine, CA, 21 May 01) (Deadline: 18 May 01)**

**(Statement A)**

1. This request has been reviewed by the Foreign Disclosure Office for: a.) appropriateness of distribution statement, b.) military/national critical technology, c.) export controls or distribution restrictions, d.) appropriateness for release to a foreign nation, and e.) technical sensitivity and/or economic sensitivity.

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APPROVED/APPROVED AS AMENDED/DISAPPROVED

PHILIP A. KESSEL  
Technical Advisor  
Space and Missile Propulsion Division

Date

# Status of CryoSolid Propellants Task

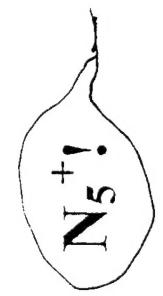
Mario E. Fajardo and Michelle E. DeRose

- \* Cryosolid Propellants Team
- \* HEDM Cryosolid Propellants Concept (Atoms in Solid Hydrogen)
- \* Cryosolid Propellants Payoffs, Objectives, Approach
- \* Requirement for Spectroscopic Diagnostics
- \* Rapid Vapor Deposition of Thick Parahydrogen ( $pH_2$ ) Solids
- \* Update on Al/ $pH_2$  and Mg/ $pH_2$  Experiments
- \* Opportunities for Supporting In-House Effort
- \* Recommendations for Future Experiments
- \* Open Discussion

# Cryosolid Propellants Team

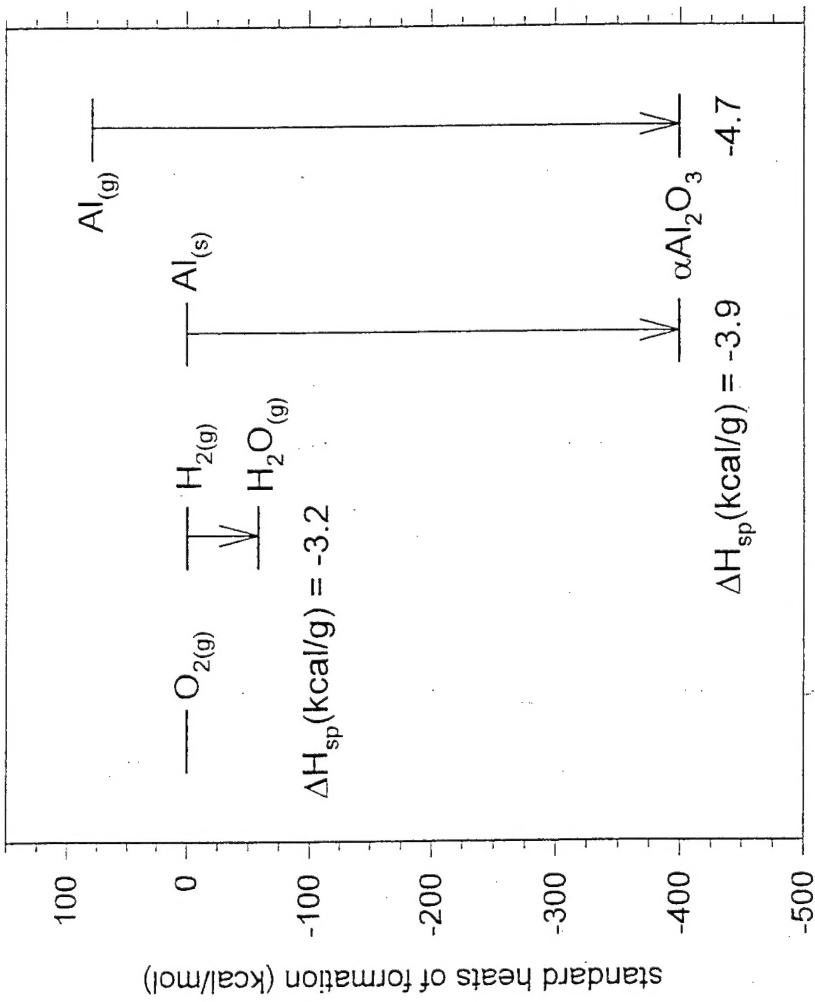
- \* Mario E. Fajardo, Michelle E. DeRose, and Simon Lam  
Bill Larson and Jessica Knappe (B atom source)
- \* J. Boatz, J. Mills, P. Langhoff, and J. Simek (in-house theory)
- \* FY00 Interactions with AFOSR Contractors:  
P. Dagdigan @ Johns Hopkins: Al/H<sub>2</sub> & B/H<sub>2</sub> Complexes  
M. Alexander @ U. Maryland: B/H<sub>2</sub> Interaction Potentials  
G. Voth @ U. Utah: Path-Integral Monte Carlo Simulations  
G. Scoles & K. Lehmann @ Princeton U.: Helium Clusters
- \* External Collaborators:  
T. Momose @ Kyoto U.: High Resolution IR Spectroscopy
- \* Summer Visiting Professors:  
R.J. Hinde @ U. Tennessee: Dopant-Induced IR Activity  
D. Anderson @ U. Wyoming: Dopant IR Absorptions

# “Revolutionary” vs. “Evolutionary” HEDM Concepts

- \* “Revolutionary” means better than LOX/LH<sub>2</sub>:
  - LOX/LH<sub>2</sub>       $\Delta H_{sp} = 12.6 \text{ MJ/kg}$  (3.0 kcal/g)
  - HEDM Target:       $\Delta H_{sp} > 15.0 \text{ MJ/kg}$  (3.6 kcal/g)
- \* Early (c1990) Revolutionary HEDM Concepts:
  - tetrahydrogen (H<sub>4</sub>)
  - metastable triplet helium (He\* and He<sub>2</sub>\*)
  - spin-polarized atomic hydrogen (H↑)
  - high-spin species (<sup>5</sup>CO)
  - dications (AB<sup>++</sup>, ABC<sup>++</sup>)
- \* “non-metallics” (e.g. O<sub>4</sub>/H<sub>2</sub>, N<sub>4</sub>, N<sub>8</sub>, N<sub>20</sub>)  **N<sub>5</sub><sup>+</sup>!**
  - metallic hydrogen
  - metal atoms and clusters in solid H<sub>2</sub>

# Cryosolid Propellants Concept

Use cryogenic solid hydrogen as a “packaging material” to store energetic species such as metal atoms and clusters.



# Cryosolid Propellants Payoffs

## Increased Specific Impulse

$$I_{sp} \propto \sqrt{\Delta H_{sp}}$$

- LOX/LH<sub>2</sub>: I<sub>sp</sub> = 400 s
- 5% B/sH<sub>2</sub> + LOX: I<sub>sp</sub> = 500 s (+25%)\*
- 5% Al/sH<sub>2</sub> + LOX: I<sub>sp</sub> = 450 s (+12%)\*

\* calculated for P<sub>chamber</sub> = 1000 PSIA, P<sub>exhaust</sub> = 14.7 PSIA

## Greater Propellant Density

- liquid H<sub>2</sub> @ 20 K: ρ = 0.070 g/cm<sup>3</sup>
- solid H<sub>2</sub> @ 2 K: ρ = 0.087 g/cm<sup>3</sup> (+25%)
- 50/50 liquid He/solid H<sub>2</sub>: ρ = 0.105 g/cm<sup>3</sup> (+50%)

# Atom Additive Payoffs (5 % molar)

Sea level specific impulse,  $I_{sp}$ , in seconds (% change)

$$P_{\text{chamber}} = 1000 \text{ PSIA}, P_{\text{exhaust}} = 14.7 \text{ PSIA}$$

| <u>Additive</u> | in standard state<br><u>M(5%)/LOX/H<sub>2</sub></u> | as atoms<br><u>M(5%)/LOX/H<sub>2</sub></u> | monoprop.<br><u>M(5%)/H<sub>2</sub></u> |
|-----------------|---|--|---|
| none            | 403   |  |   |
| C               | 381 (-5%)   | 515 (+28%)                                 | 515 (+28%)                              |
| B               | 407 (+1%)   | 508 (+26%)                                 | 465 (+15%)                              |
| Be              | 427 (+6%)   | 493 (+22%)                                 |   |
| Si              | 400 (-1%)   | 460 (+14%)                                 |   |
| Al              | 407 (+1%)   | 454 (+13%)                                 |   |
| H               | 403   | 430 (+7%)                                  | 380 (-6%)                               |
| Li              | 404   | 428 (+6%)                                  |   |
| Mg              | 400 (-1%)   | 416 (+3%)                                  |   |

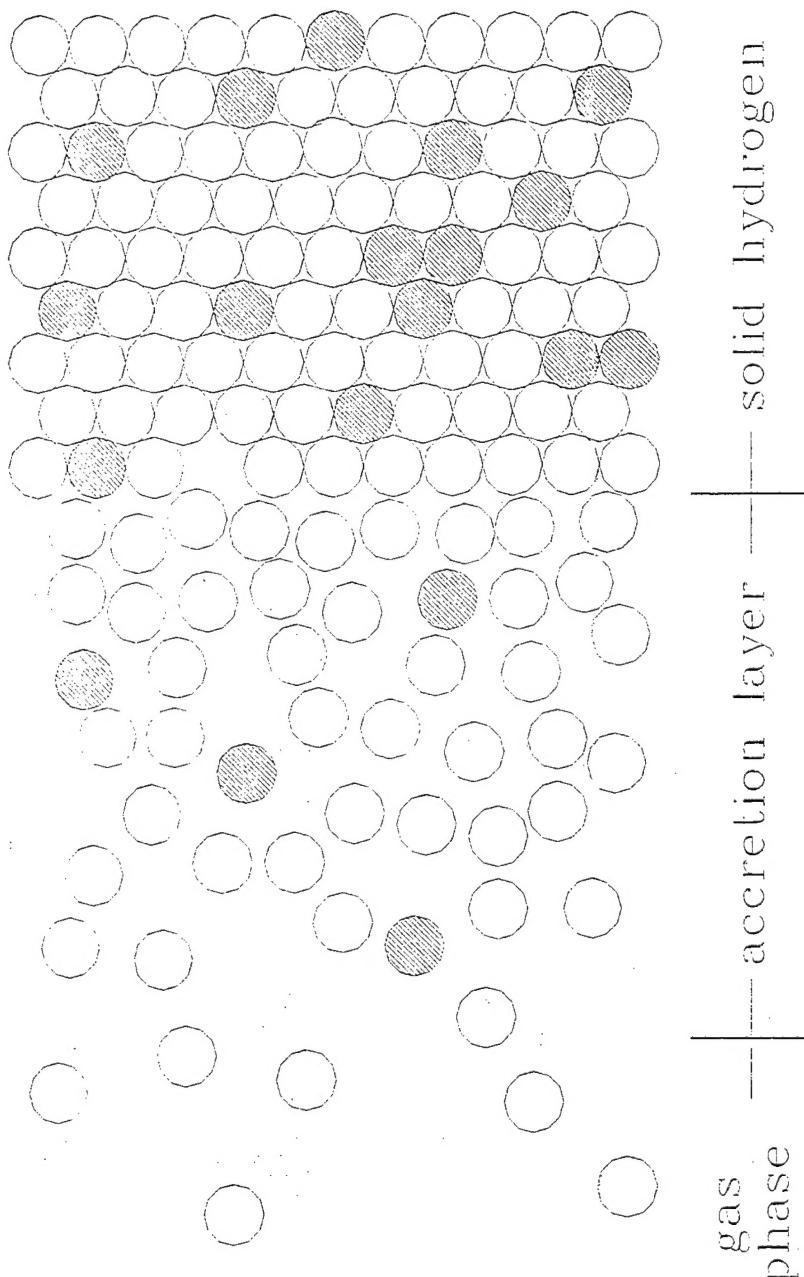
# Cryosolid Propellants Objectives

- \* Make solid hydrogen samples (any size) containing 5% molar concentration of trapped energetic additives.
- \* Measure absolute concentrations of energetic species.
- \* Scale-up samples; produce  $\sim 1 \text{ cm}^3$  samples in our lab.

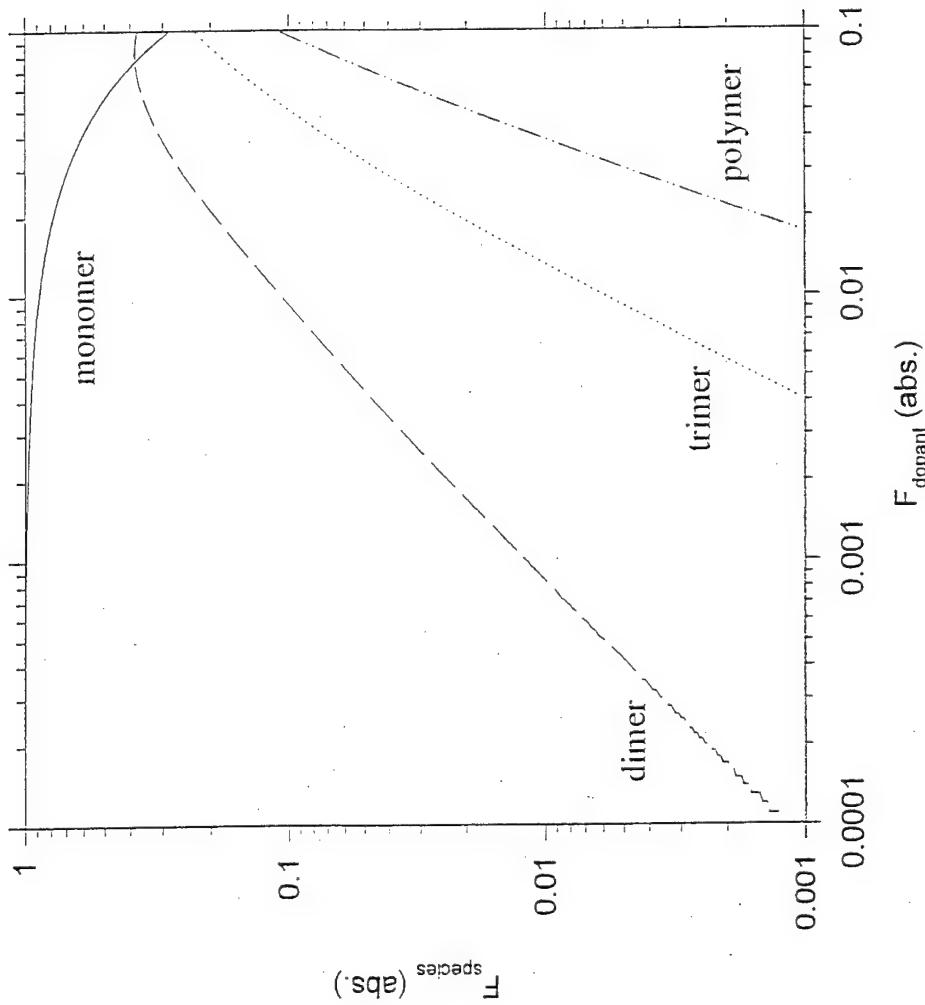
Example: 5% Al/pH<sub>2</sub>, V = 1 cm<sup>3</sup>  
assume each Al atom replaces one H<sub>2</sub> molecule  
 $\Rightarrow$  58 mg Al / 83 mg H<sub>2</sub> (\*see display item\*)  
 $\therefore \rho = 0.142 \text{ g/cm}^3 (+100\%)$

# Cryosolid Propellants Approach

- \* Rapid vapor deposition of metal atom vapor and pre-cooled parahydrogen gas onto a liquid helium cooled substrate in vacuum.



# Recombination at High Concentrations



Bernoulli Trails (statistical) model of dopant agglomeration:  $P(k, 12) = \binom{12}{k} f^k (1-f)^{12-k}$

# Requirement for Spectroscopic Diagnostics

- \* Develop spectroscopic techniques to identify and measure concentrations of trapped species.
- \* Beer's Law:  $A = \sigma N d$ 
  - $A$  = absorbance =  $-\ln(I/I_0)$
  - $\sigma$  = absorption cross-section
  - $N$  = species number densities
  - $d$  = pathlength
  - $N d$  = "column density"
- \* UV/vis absorption for low metal atom column densities.
- \* IR absorptions, direct and dopant-induced pH<sub>2</sub> transitions, for reaction products & metal atoms at large column densities.

# The Perils of Calorimetry

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GEORGE C. PIMENTEL

TABLE IX  
CONCENTRATIONS OF FREE RADICALS REPORTED

| Radical                | Matrix                              | Mole per cent radicals | Method of production and estimate <sup>a</sup> | Reference                       |
|------------------------|-------------------------------------|------------------------|--|---------------------------------|
| O <sub>2</sub>         | O <sub>2</sub>                      | 4-20                   | Gas, cal                                       | Minkoff <i>et al.</i> (1959).   |
|                        |                                     | <3                     | Gas, IR  | Havroy and Bass (1958)          |
|                        |                                     | ~1                     | Gas, cal                                       | Broida and Lutes (1950)         |
| O <sup>+</sup>         | C <sub>6</sub> (OH) <sub>2</sub>    | 0.0                    | γ, ESR   | R. Livingston <sup>b</sup>      |
| N <sup>+</sup>         |                                     | 4                      | Gas, cal                                       | Minkoff <i>et al.</i> (1959)    |
|                        |                                     | 0.2                    | Gas, cal                                       | Broida and Lutes (1950)         |
|                        |                                     | 0.03                   | γ, ESR   | Wall <i>et al.</i> (1959b)      |
|                        |                                     | >0.03                  | Gas, cal                                       | Fontana (1958)                  |
|                        |                                     | 0.01-0.04              | Gas, MS  | Fontana <sup>c</sup>            |
| OH(?)                  | HCOOH                               | 0.2                    | γ, ESR   | Matheson and Smuller (1955)     |
| CH <sub>3</sub>        | CH <sub>4</sub>                     | 0.14                   | γ, ESR   | Wall <i>et al.</i> (1959a)      |
| H                      | CH <sub>4</sub>                     | 0.1                    | γ, ESR   | Wall <i>et al.</i> (1959a)      |
| N                      | NH <sub>3</sub>                     | 0.1                    | Gas, ESR                                       | Cole and Harding (1958)         |
| H                      | HClO <sub>4</sub> -H <sub>2</sub> O | 0.1                    | γ, ESR   | Livingston <i>et al.</i> (1955) |
| H                      | H <sub>2</sub> O                    | 0.01                   | γ, ESR   | Matheson and Smuller (1955)     |
| H, NH <sub>2</sub> (?) | NH <sub>3</sub>                     | 0.01                   | UV, ESR  | D. Ingram <sup>b</sup>          |
| ROH                    | Alcohols                            | ~0.01                  | UV, ESR  |                                 |
| H                      | H <sub>2</sub>                      | 0.0006                 | γ, ESR   | Wall <i>et al.</i> (1959a)      |

<sup>a</sup> Abbreviations: gas = rapid condensation of gaseous radicals; γ = gamma ray *in situ* production; UV = photolytic *in situ* production; IR = infrared analysis; cal = calorimetry; MS = magnetic susceptibility.

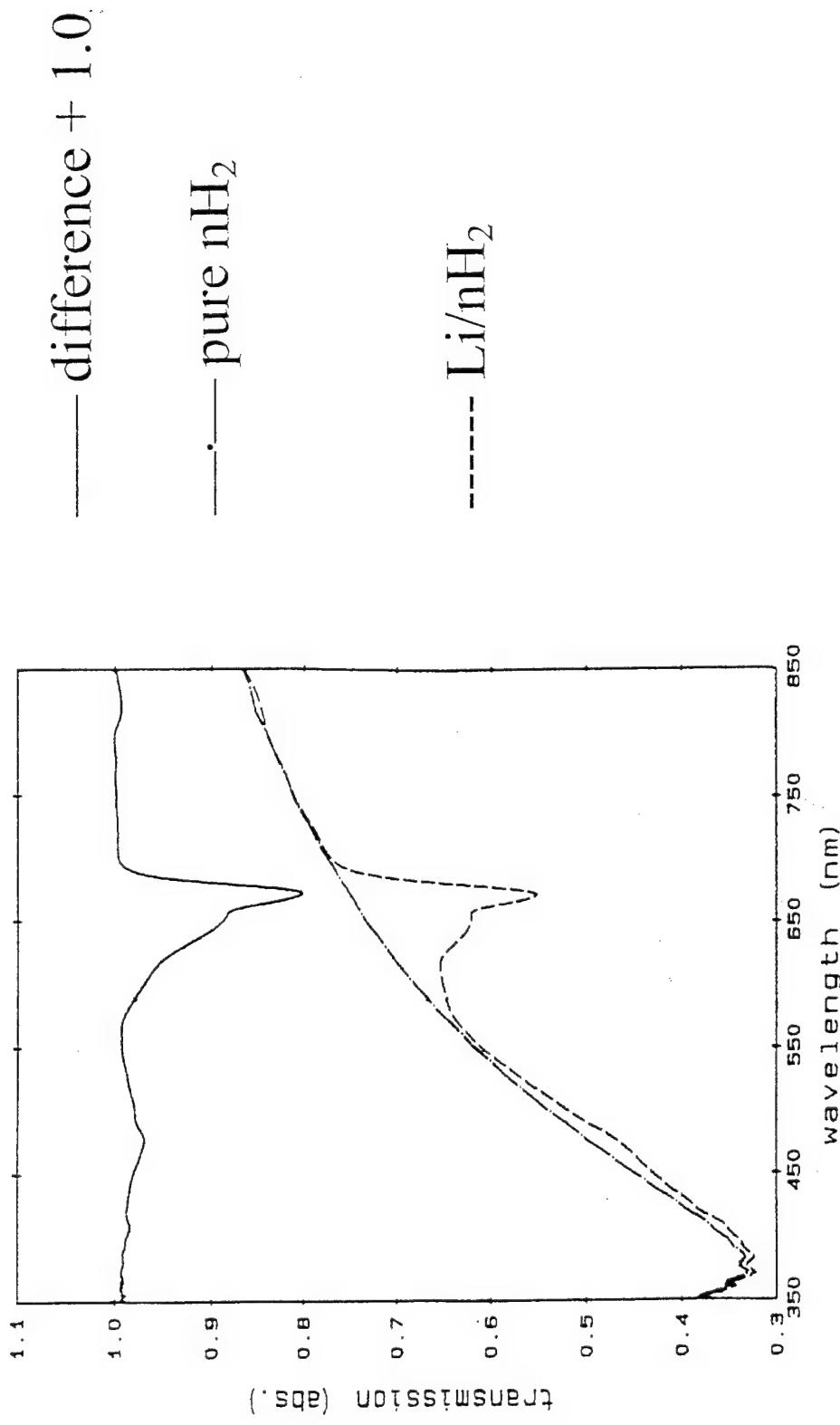
<sup>b</sup> Private communication.

<sup>c</sup> Fontana, B. J. (1959). *J. Chem. Phys.* **31**, 148.

- \* Difficult to distinguish energy release by small concentrations of very energetic species vs. low energy re-arrangements of host.

[A.M. Bass and H.P. Broida, "Formation and Trapping of Free Radicals" (Academic, New York, 1960)]

# Transmission Spectrum of Li/nH<sub>2</sub>, d ≈ 10 μ



M.E. Fajardo, J. Chem. Phys. **98**, 110 (1993).

# Optical Scattering in Solid Hydrogen

## Crystal Growing and Quality (p. 81)

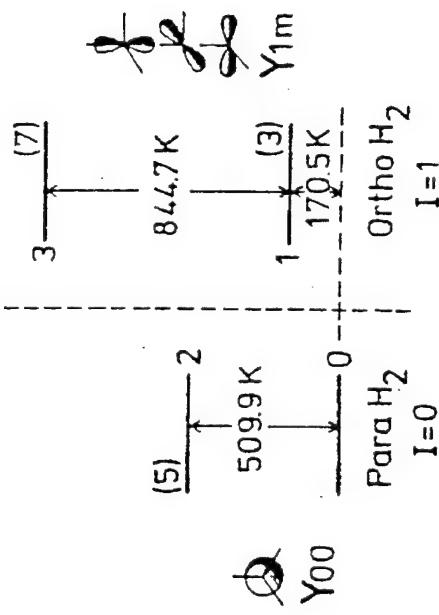
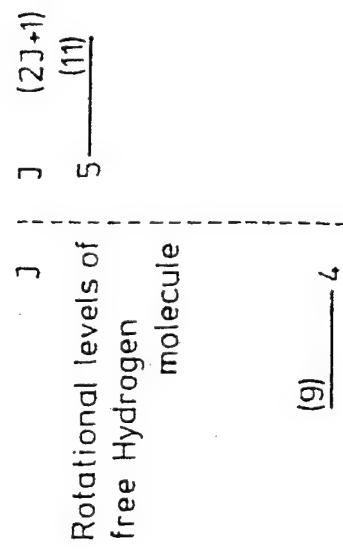
“There is a considerable art to growing hydrogen crystals of high quality. Good crystals are always grown slowly from the melt; a rapid freeze from the gas produces snow.”

## Crystallite Light Scattering (p. 83)

“The reason that a good hydrogen crystal is so hard to see is its low refractive index...an estimated 1.16!

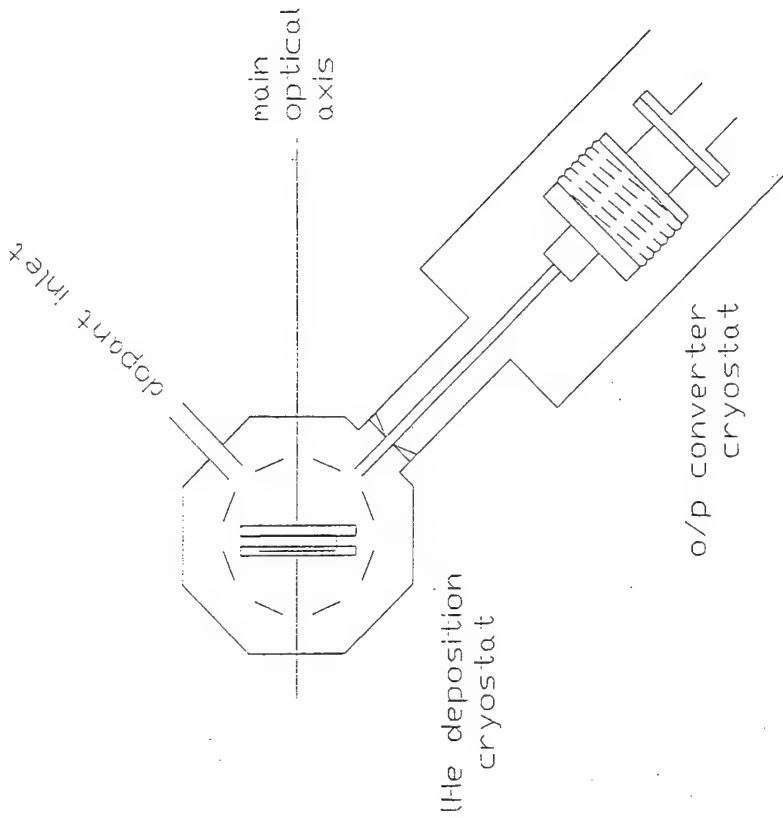
Yet a 1 mm-thick layer of hydrogen crystallites can be a completely opaque brown-black.”

# ortho- and para-hydrogen



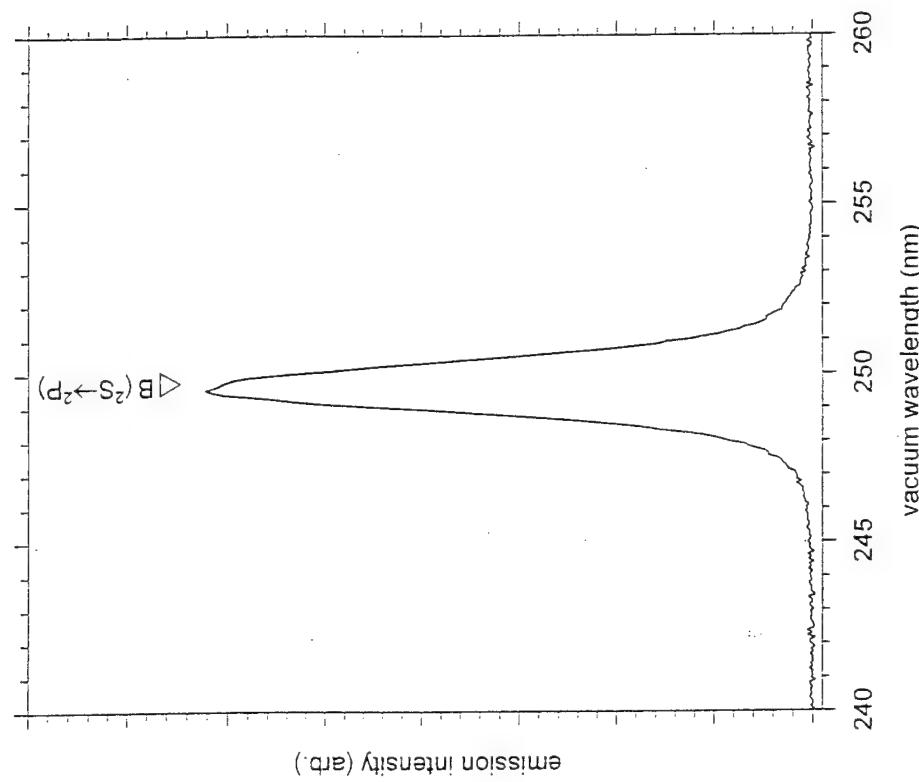
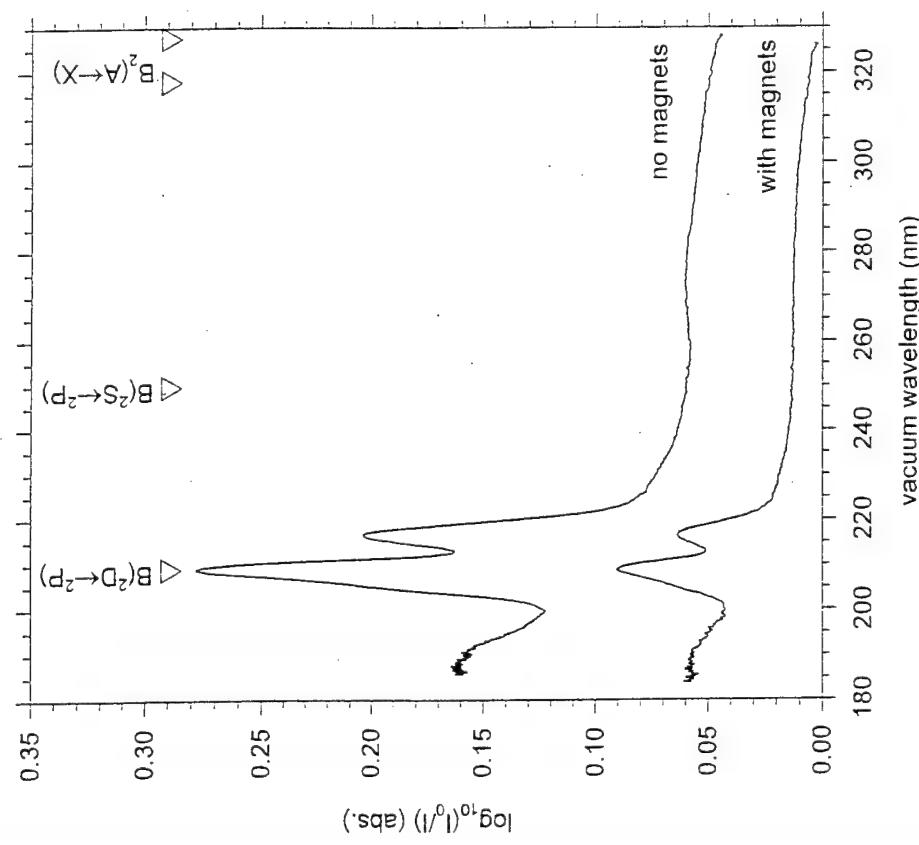
[I.F. Silvera, Rev. Mod. Phys. **52**, 393 (1980)]

# Rapid Vapor Deposition of Gram-Scale Optically Transparent pH<sub>2</sub> Solids (c1997)



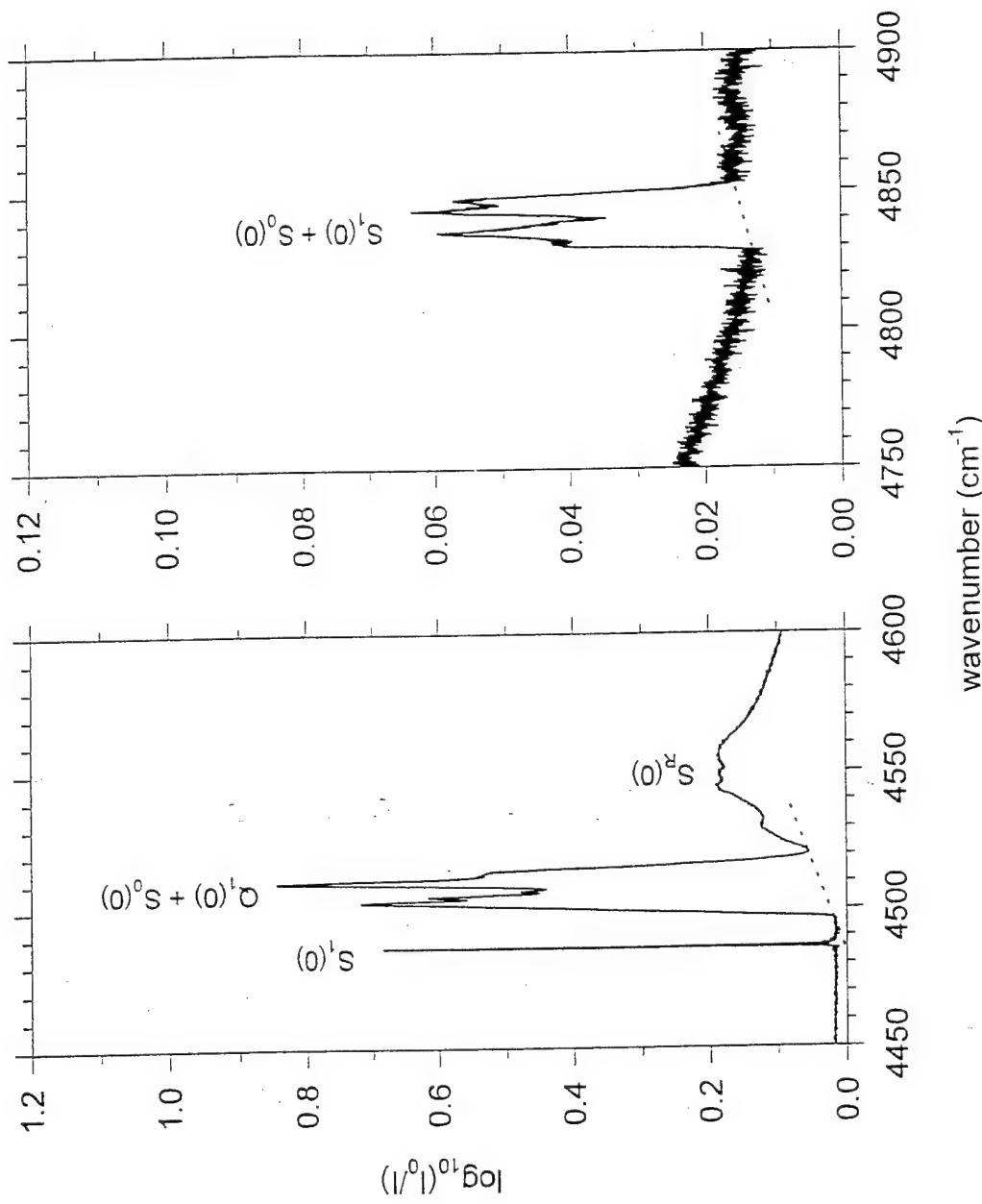
M.E. Fajardo and S. Tam, J. Chem. Phys. **108**, 4237 (1998).  
S. Tam and M.E. Fajardo, Rev. Sci. Instrum. **70**, 1926 (1999).

# Electronic Spectroscopy of B/pH<sub>2</sub> (d≈2 mm)



S. Tam, M. Macler, M.E. DeRose, and M.E. Fajardo, J. Chem. Phys. **113**, 9067 (2000).  
[J.R. Krumrine, S. Jang, G.A. Voth, and M.H. Alexander, J. Chem. Phys. **113**, 9079 (2000)]

# Solid pH<sub>2</sub> Thickness from IR Spectra



S. Tam and M.E. Fajardo, Rev. Sci. Instrum., submitted (2001).

# Absorption Intensity VS. Thickness

new values:

$$\int A_{Q+S} dV / d_{\text{total}} = 82 \pm 2 \text{ cm}^{-2}$$

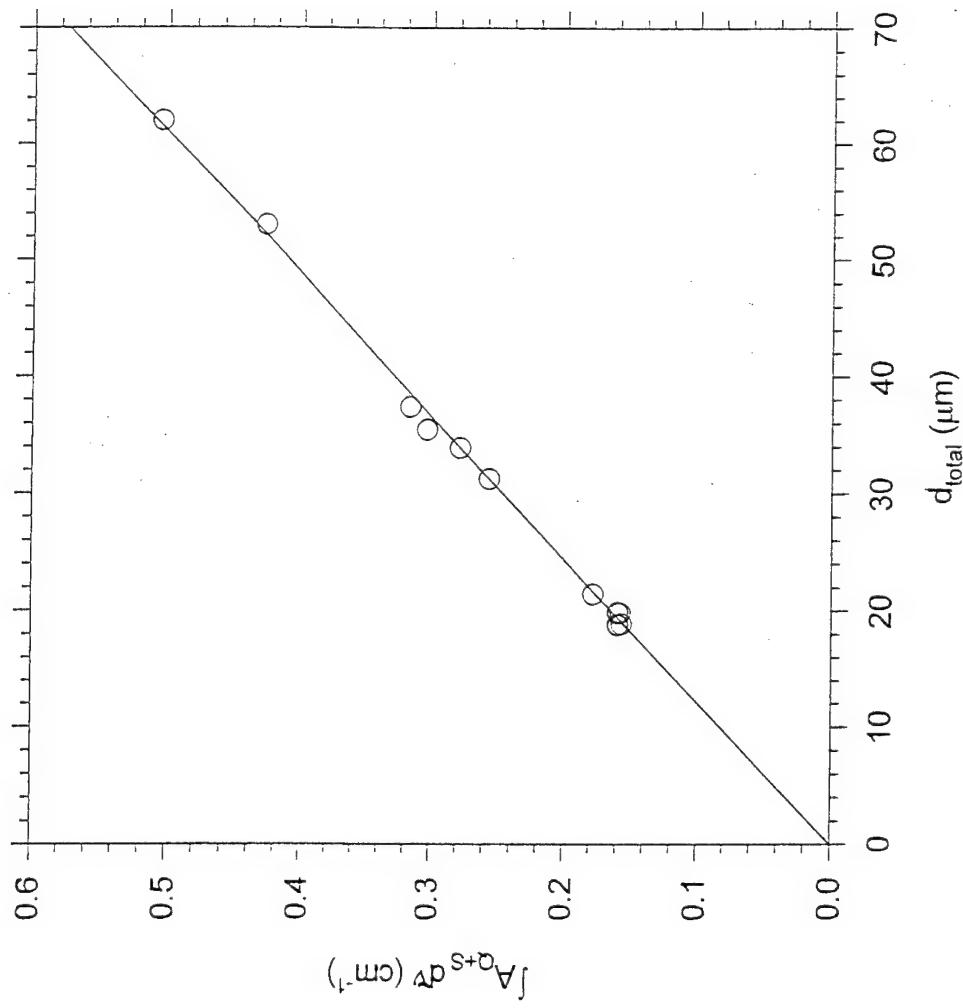
$$\int A_{S+S} dV / d_{\text{total}} = 6.3 \pm 0.3 \text{ cm}^{-2}$$

$$\tilde{\alpha}_{Q+S} = 4.84(\pm 0.13) \times 10^{-14} \text{ cm}^3/\text{s}$$

$$\tilde{\alpha}_{S+S} = 0.35(\pm 0.02) \times 10^{-14} \text{ cm}^3/\text{s}$$

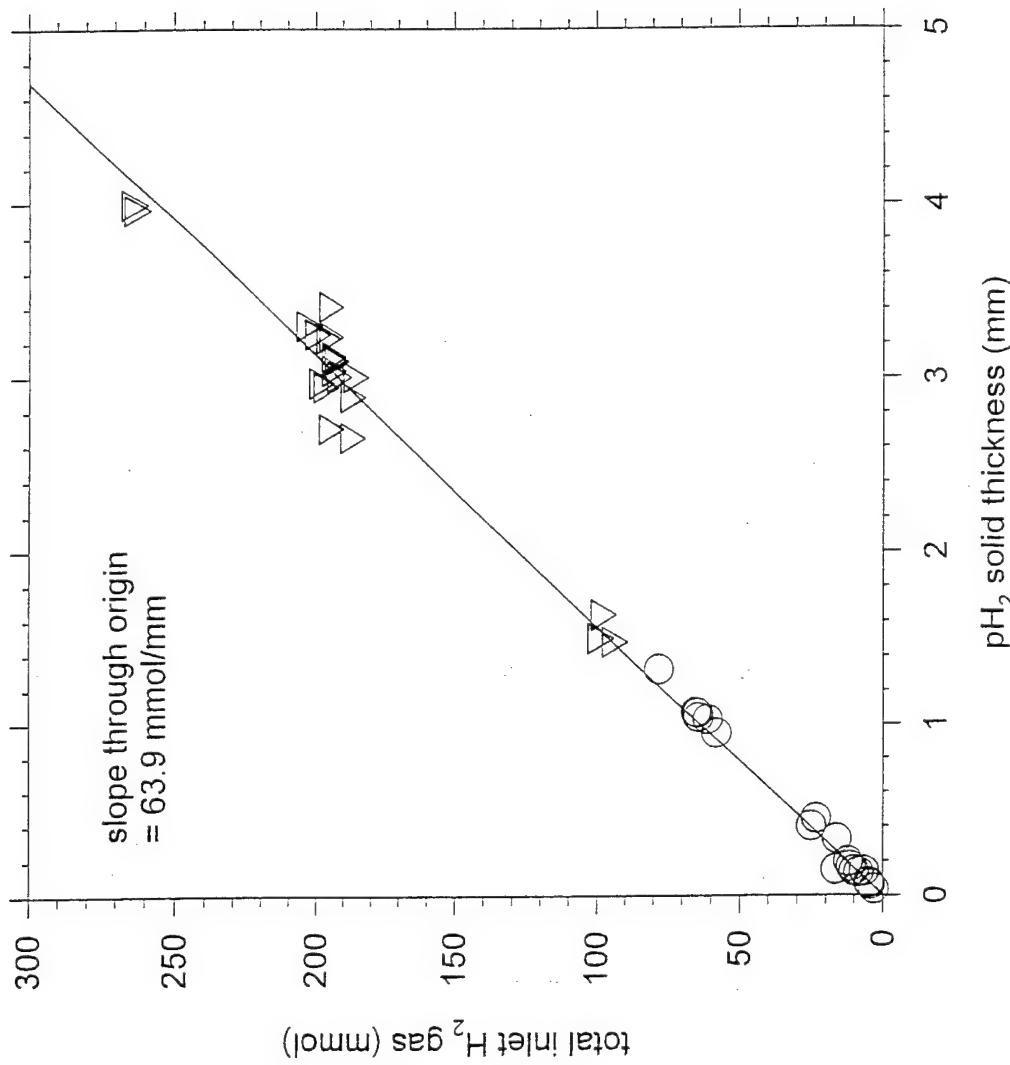
literature (c1960):

$$\tilde{\alpha}_{Q+S} = 4.5(\pm ?) \times 10^{-14} \text{ cm}^3/\text{s}$$



S. Tam and M.E. Fajardo, Rev. Sci. Instrum., submitted (2001).

# Constant pH<sub>2</sub> Deposition Efficiency



# High Flux HEDM Sources

- \* Purchased commercial Al evaporator; PBN crucible holds  
 $\approx 10 \text{ g Al}$  in horizontal orientation.
- \*  $T_{\max} = 1200 \text{ }^{\circ}\text{C} \Rightarrow P_{\text{vap}}(\text{Al}) \approx 8 \times 10^{-3} \text{ torr} \Rightarrow \Phi_{\text{Al}} \approx 10^{18} \text{#/cm}^2\text{-s}$

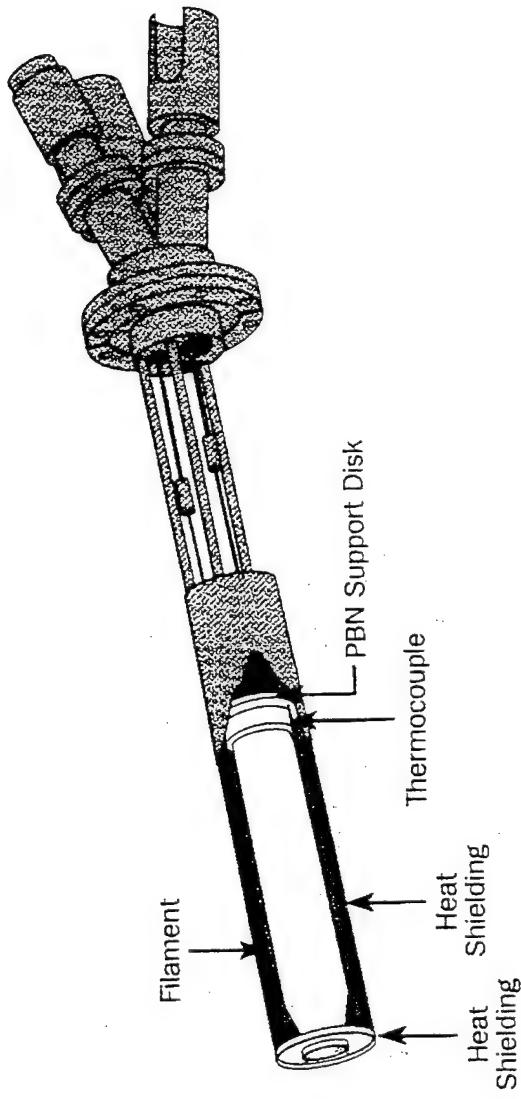
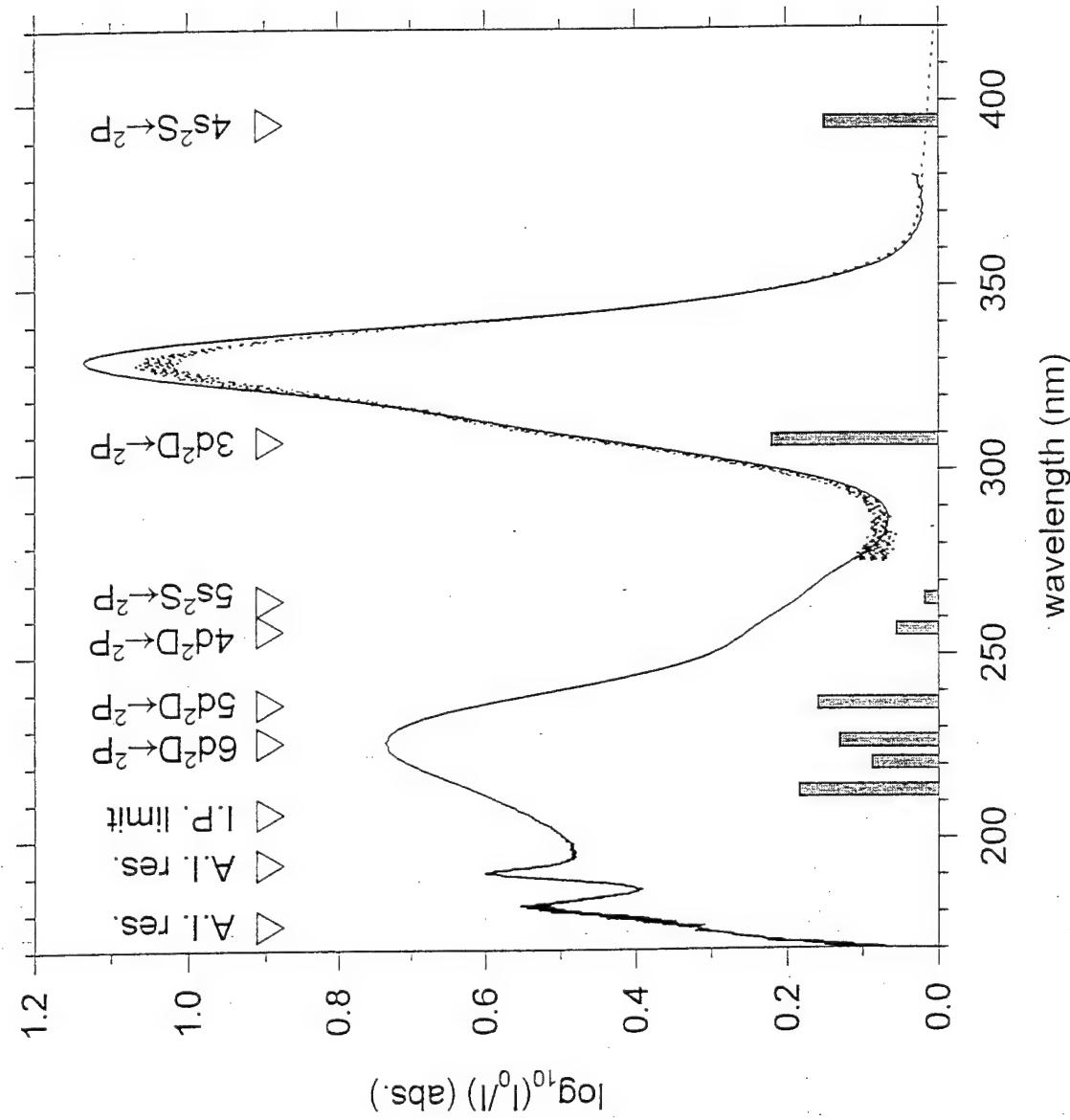
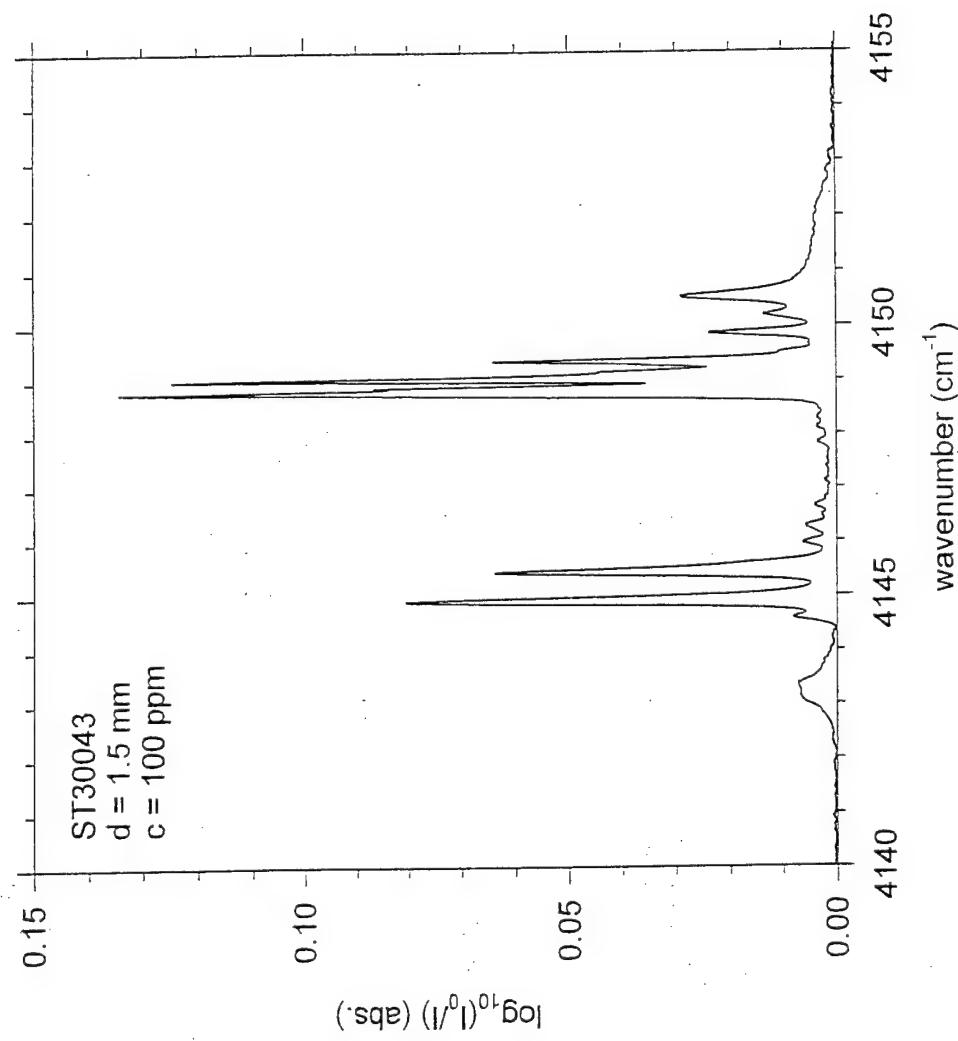


Figure 1-3: Schematic of the EPISUMOM Effusion Cell.

# 60 ppm Al/pH<sub>2</sub> UV Absorption (d=0.14 mm)

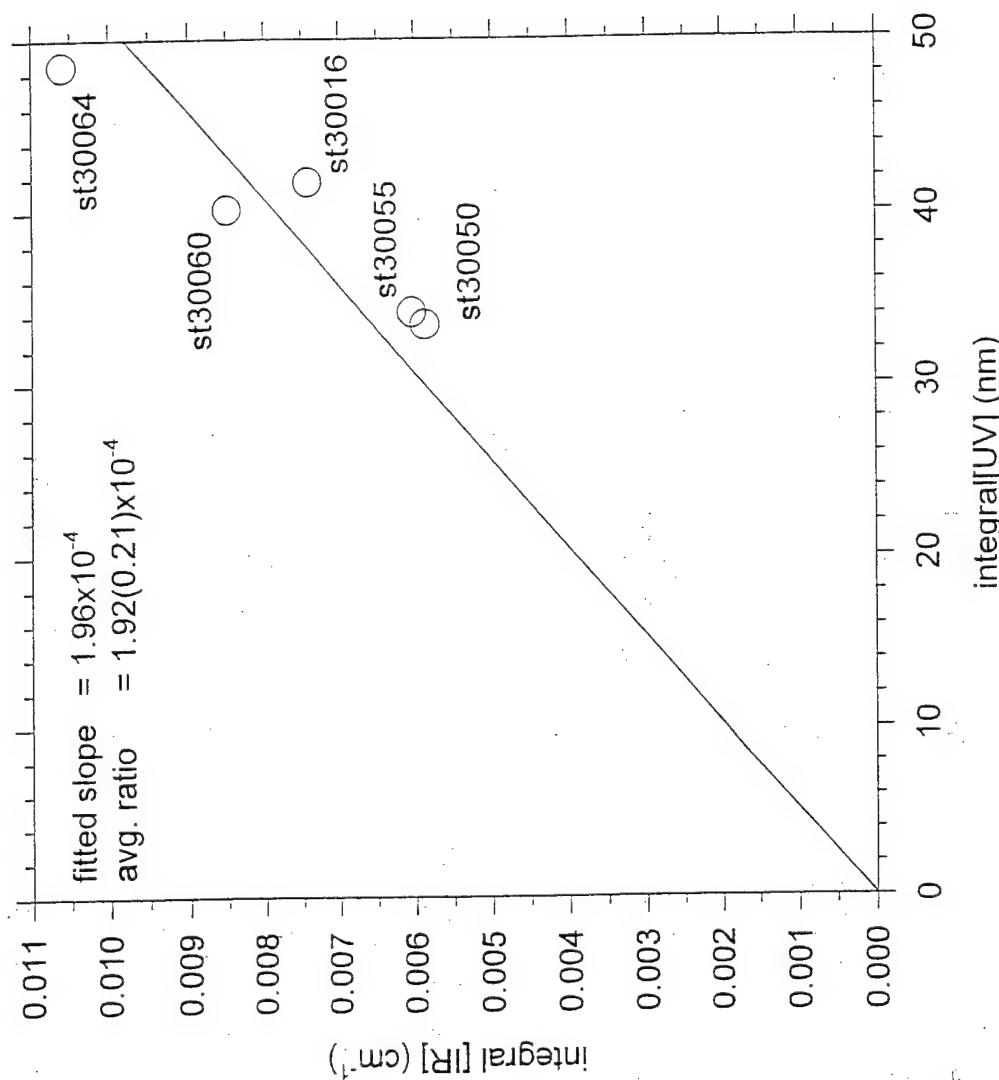


# Al-induced IR Absorption Spectrum

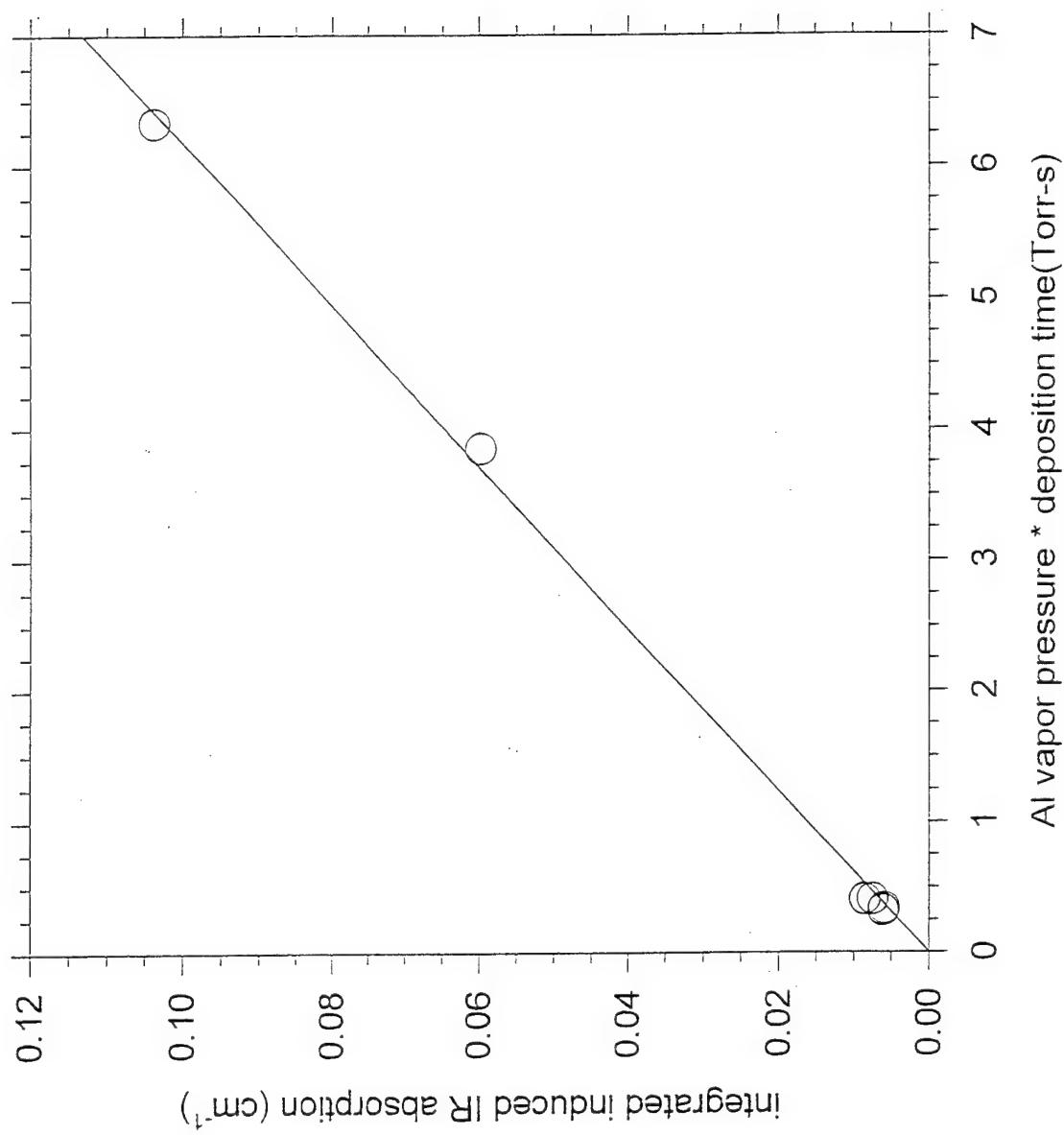


Analysis in collaboration with Prof. R.J. Hinde, U. Tennessee (Knoxville).

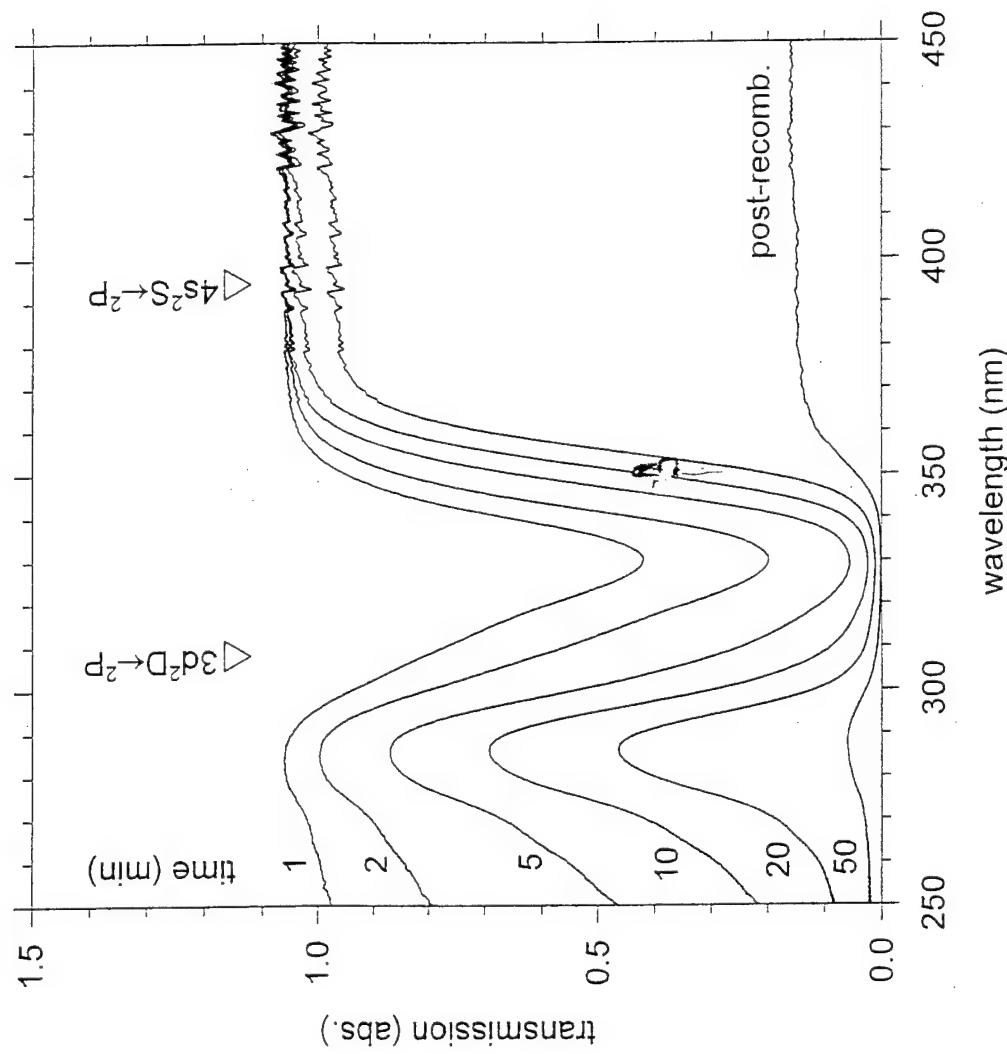
# Correlation Between UV & IR Absorptions



# Constant Al Atom Deposition Efficiency



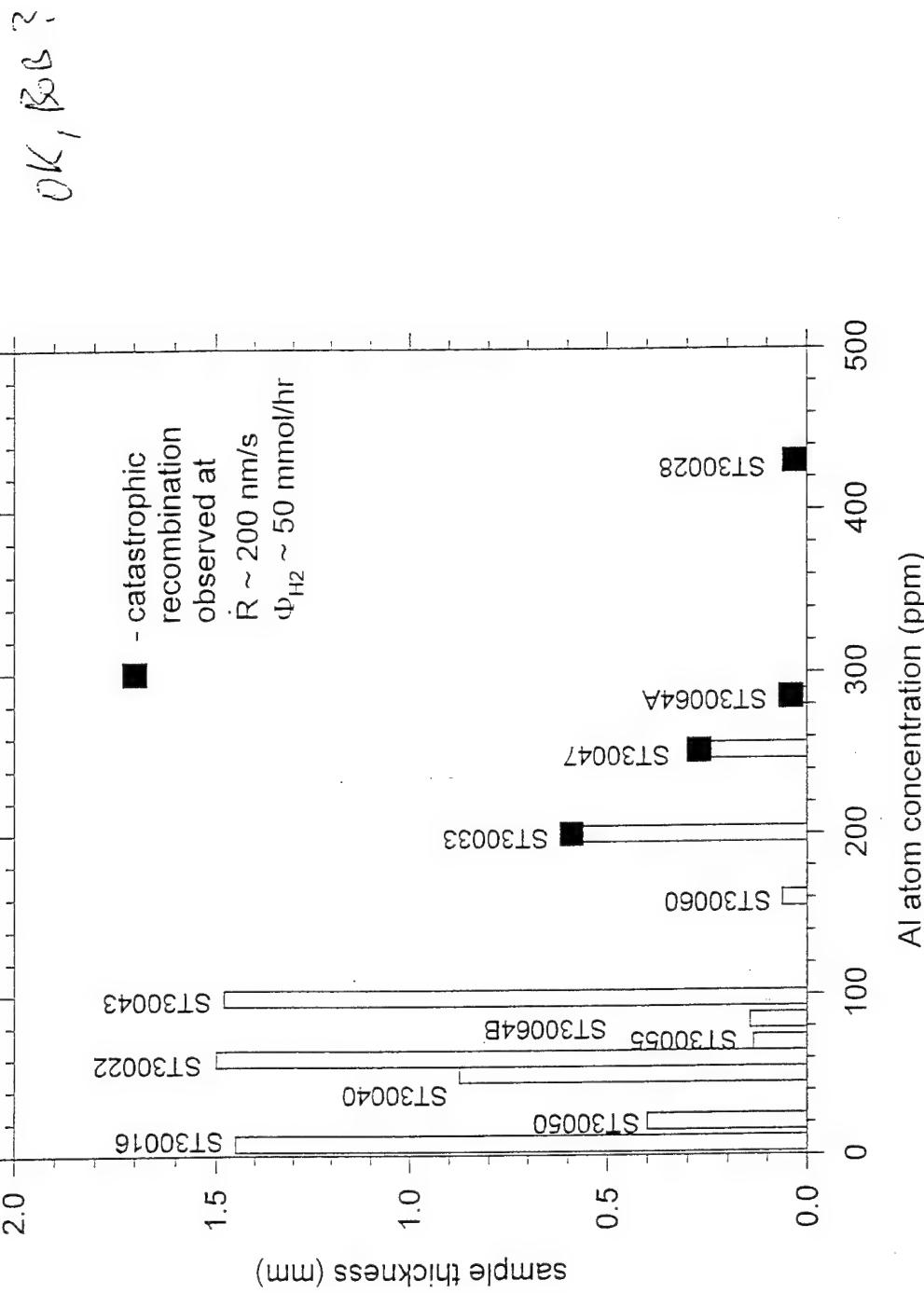
# UV Spectrum of Recombined Al/pH<sub>2</sub>



# Recombination/reaction in Al/pH<sub>2</sub>

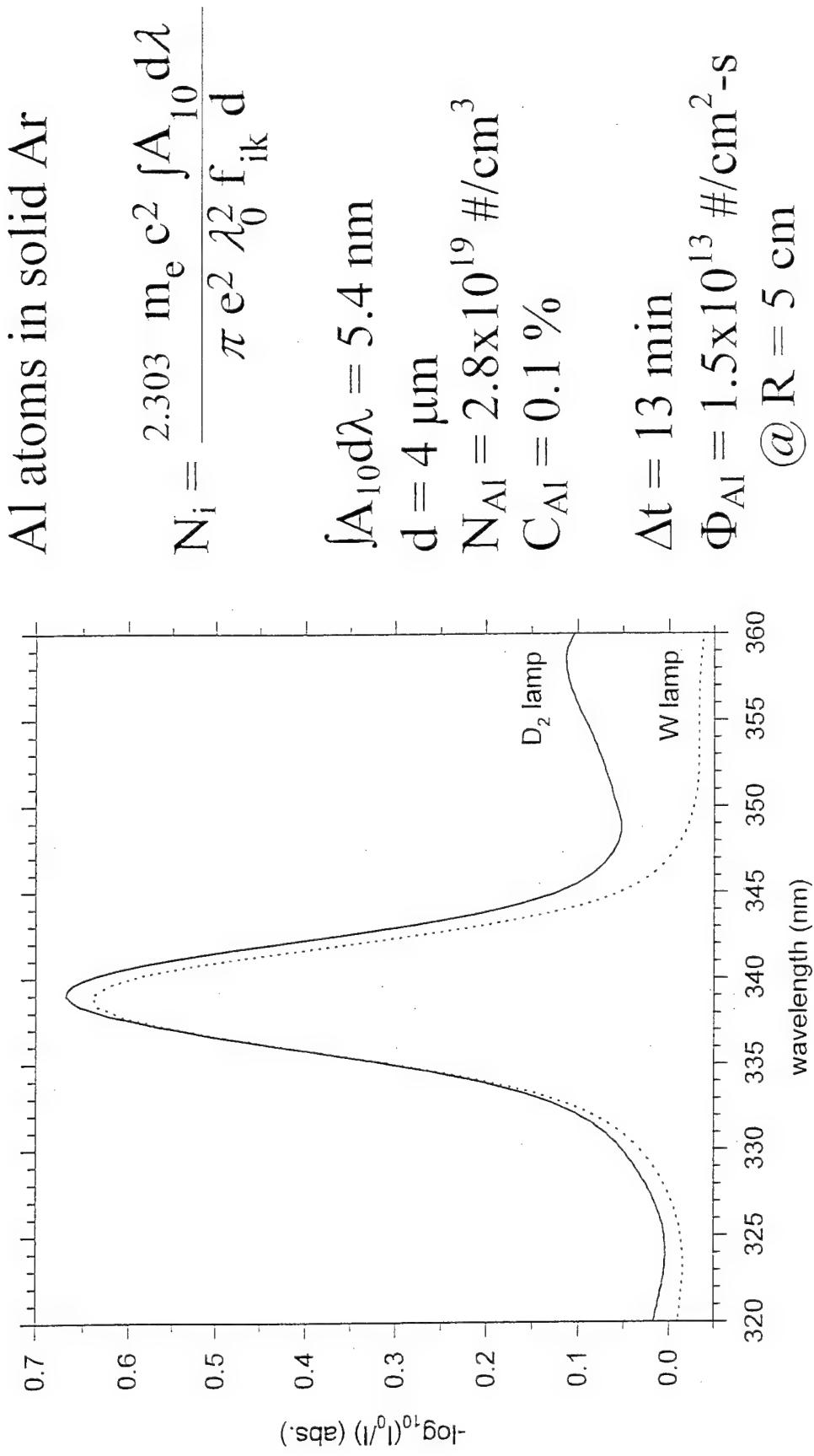


# Summary of Al/pH<sub>2</sub> Deposition Results



# 1000 ppm Al/Ar

- \* Evaporate Al from 1 mm dia. Ta filaments



## Al/pH<sub>2</sub> Summary

- \* Demonstrated trapping of  $\approx 0.1\%$  Al atoms in thin Ar solids using home-made Al atom source.
- \* Demonstrated Al-induced IR absorption by pH<sub>2</sub> molecules as a diagnostic of Al atom concentrations in high column density samples.
- \* Attempts (four to date) to exceed 0.02 % Al atom concentrations in thick pH<sub>2</sub> solids failed, resulting in catastrophic recombination (and reaction?) of the Al atoms.
- \* Interpretation: thick vapor deposited pH<sub>2</sub> solids impede dissipation of heat released upon atomic recombination, causing a recombination cascade. Attempt depositions of thin ( $\sim 10\ \mu\text{m}$ ), high Al atom concentration Al/pH<sub>2</sub> samples (worked for Ar matrices).

# Is Catastrophic Recombination Ubiquitous to Rapid Vapor Deposited pH<sub>2</sub> Solids?

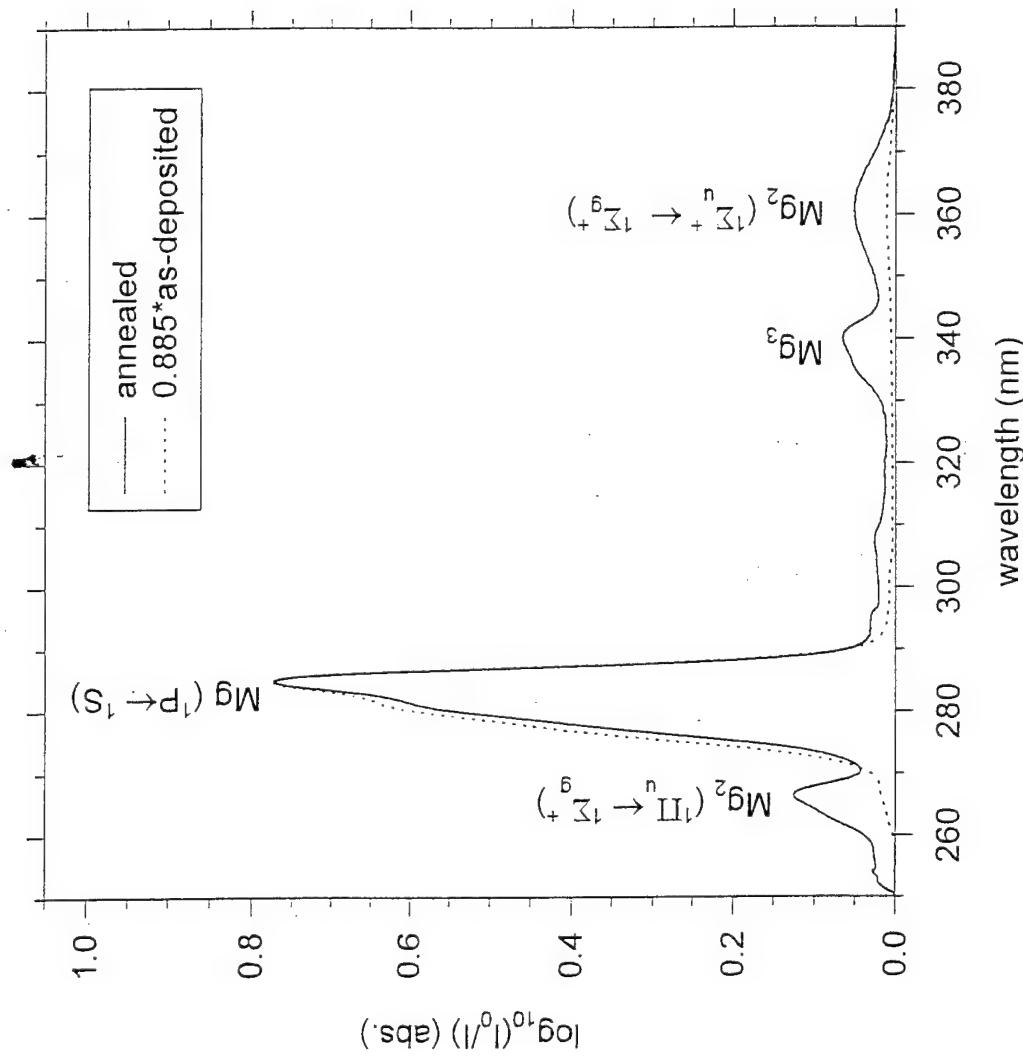
- \* 
$$\frac{1}{k_{\text{recomb}}} = \frac{1}{k_{\text{intrin}}} + \frac{1}{k_{\text{diff}}}$$
- \*  $k_{\text{intrin}}$  depends on M-M interaction potential,  
increases with increasing well depth and range
- \*  $\Rightarrow$  try Mg atoms as "HEDM" dopant  
Mg-Mg and Mg-Mg<sub>n</sub> "van der Waals" interactions

# M-M Diatomic Ground State Potentials

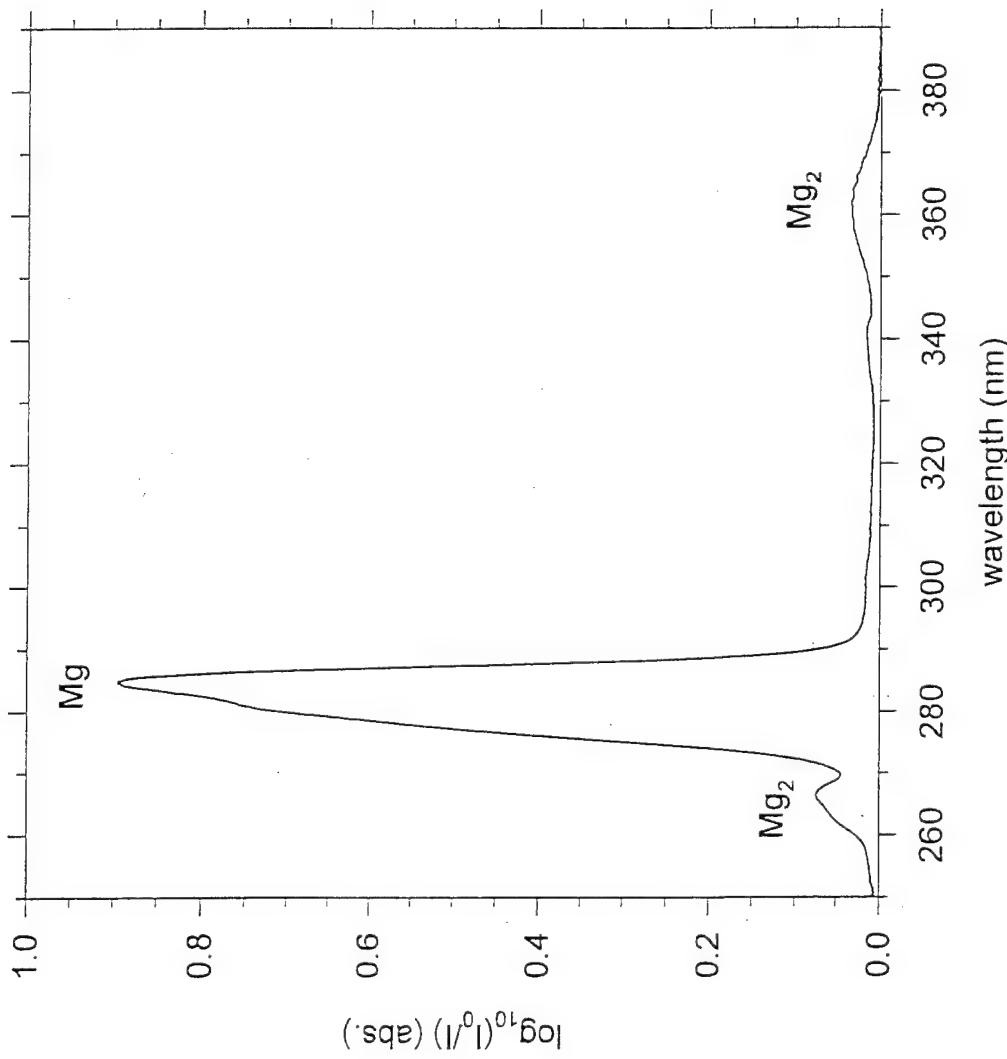
\*\*\*\*\*INCOMPLETE SLIDE\*\*\*\*\*

- \* Will make PLOT comparing Al-Al and Mg-Mg ground state potentials, available from open literature.
- \* if not too cluttered, include Li-Li, B-B potentials, too.
- \* shallower wells and especially weaker long-range interactions mean reduced intrinsic recombination rates, according to G. Voth.

# Annealing of 10 ppm Mg/pH<sub>2</sub> (d=0.07 mm)



# As-Deposited 35 ppm Mg/pH<sub>2</sub> (d=0.02 mm)



# Contractor Support of In-House Effort

- \* Assignment of Al/pH<sub>2</sub> UV absorptions.  
can change calculated Al atom concentrations by factor of 2!
- \* Modeling of Al atom induced IR absorptions
- \* Modeling of Mg recombination in solid pH<sub>2</sub> (?)
- \* Open Discussion:  
coordination of efforts  
other suggestions?

# Recommendations for Future Experiments

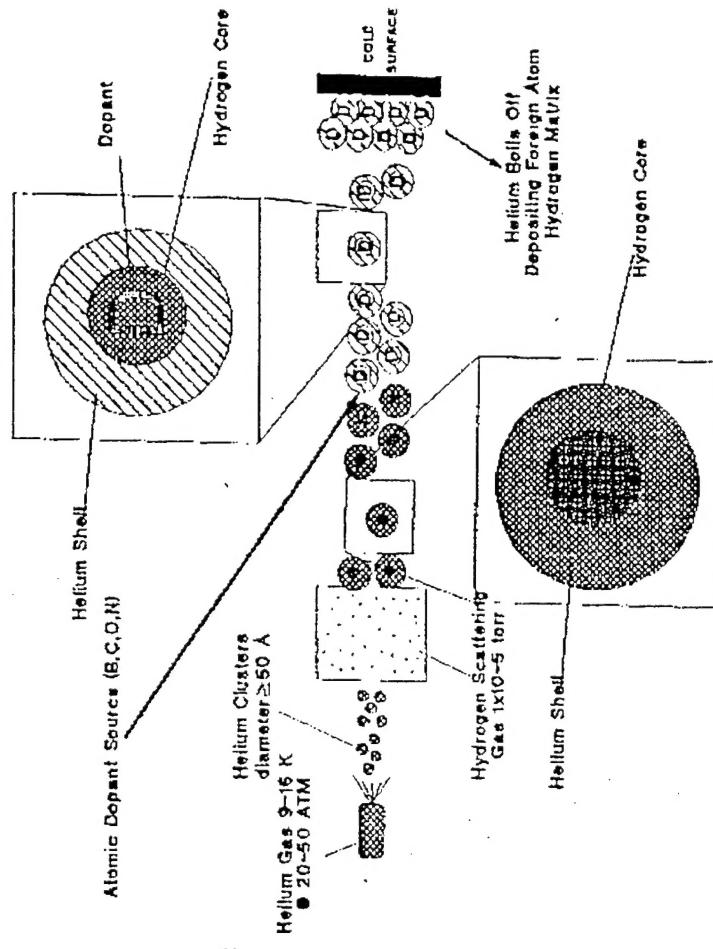
- \* In-House:
  - Complete Al/pH<sub>2</sub> and Mg/pH<sub>2</sub> experiments
  - Compare Al vs. Mg to test effects of k<sub>intrin</sub>
  - Vary pH<sub>2</sub> deposition rate/sample thickness , and compare with Ne and Ar hosts to test k<sub>diff</sub>
  - Evaluate feasibility of rapid vapor deposition method
  - Thermal B atom source SBIR project underway (B. Larson)
  
- \* AFOSR Contractors:
  - Cluster Pickup and Deposition
  - Deposition Directly onto IHe II (ala Gordon)
  - In-Situ Photolysis of HEDM Precursors (Apkarian, Stwalley)
  - Open Discussion: other suggestions?

# Future Direction: Cluster Deposition (?)

Toward the Production of Measurable Quantities of Highly Doped Solid Hydrogen

High Energy Density Matter Contractors Conference  
June 4-7, 1995

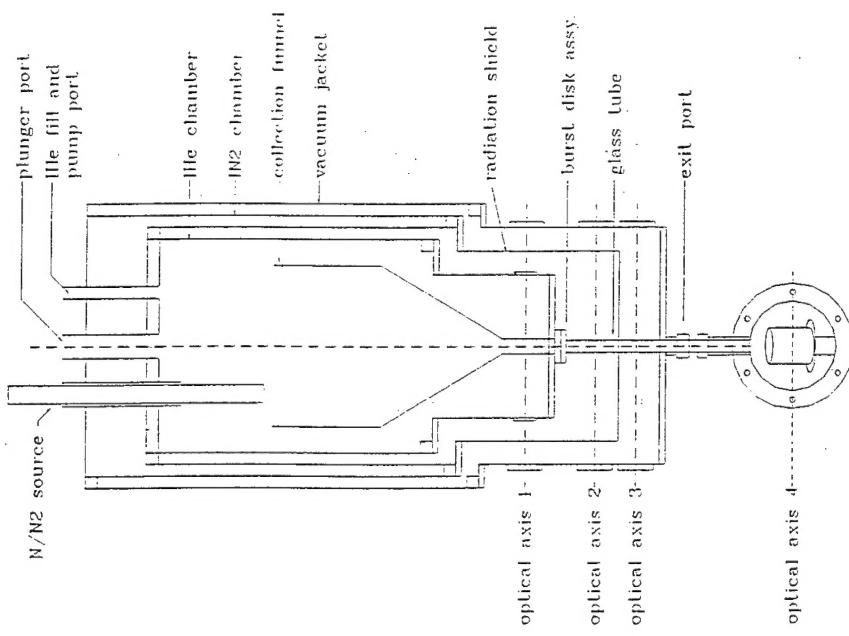
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The goal of this effort is to produce measurable quantities of highly doped (5-10%) solid hydrogen. The desired dopant atoms are C, B, O, and N. Our approach to this material is via the cluster deposition technique, as illustrated in Figure 1. First, a beam of large He clusters (between  $10^4$  and  $10^5$  He atoms per cluster) is created following the methods of Toennies<sup>1</sup> and Scoles.<sup>2</sup> Next, the He clusters are passed through a scattering chamber that contains hydrogen molecules and, in the process, "pick-up" between 12 and 20 hydrogen molecules. During the pick up process, sufficient He atoms evaporate from the cluster to maintain a temperature of about 1 K, and the hydrogen is expected to make a small micro-crystal within the He cluster. Next, the He cluster picks up the desired dopant atom. Again, He atoms evaporate to cool the cluster back to 1 K.

- \* Most excess heat is dissipated before the clusters are deposited.
- \* Chance to beat the "statistical limit" of stored atom concentration.
- \* Either higher fluxes or UHV deposition environment required.

# Future Direction: Deposition onto UHe II (?)



Based on E.B. Gordon's work

"Big-Flush" (c1995)

CESSE discharge of N<sub>2</sub>/He

Optical emission was only diagnostic  
(N/D<sub>2</sub> samples didn't glow!)

Need alternative (species specific)  
experimental diagnostics: ESR,  
NMR, FIR absorption... (?)

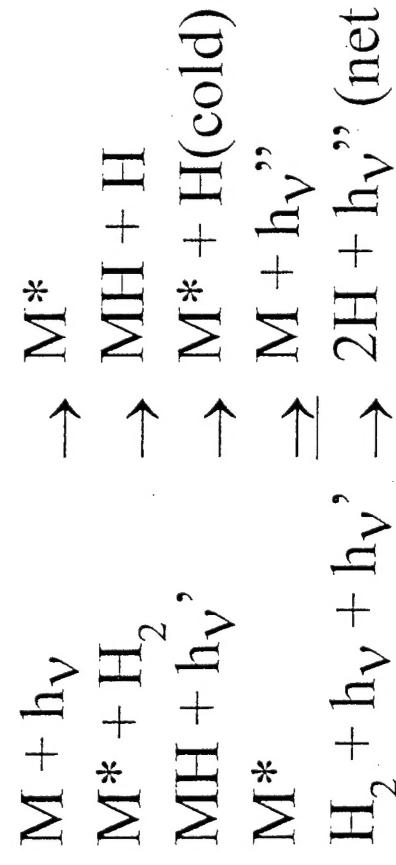
# Future Direction: In-Situ Photolysis (?)

- \* V.A. Apkarian, et al. experiments (c1994):



(5% O/H<sub>2</sub>  $\Rightarrow$  25s (6%) I<sub>sp</sub> improvement, so Al > O > H)  
UV induced desorption of H<sub>2</sub>O inside vacuum chamber  
complicates monitoring O + H<sub>2</sub> reactions.

- \* W.C. Stwalley proposal (c1992):



# Experimental Admonitions

- \* Must work with hydrogen! Some efforts on model systems are fine, but results may not generalize cleanly to hydrogen.
- \* Must focus on production and quantitative measurement of  $\sim 1\%$  concentrations of energetic species in solid hydrogen. Species specific diagnostics are preferred.
- \* Worry about scaling up later, must demonstrate progress towards larger concentrations to maintain viability of Cryosolid Propellants Task.